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DEPARTMENT OF THE ARMY
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The Electronmicroscopical Picture of the Surface
of Membrane Filters,

by
Gunter Hausmann and Helmut Pietsch.

The membrane filters of Zsigmondy and Bachmanns (reference 2) developed from the sensitive collodion ultra filters of Bechhold (reference 3) were obtained (reference 4) by dessication of nitrocellulose solutions of a determined composition in a very fine pored form with graded pore sizes. Whereas only a couple of years ago such collodion membranes could be kept only in a damp condition. (reference 5) it is possible today by suitable combination of the average solution media to produce durable filters in the dry state. We can be especially thankful to this circumstance that they could be used with success for bacteriologic and hygienic investigations. (reference 6).

The Zsigmondy filters are distinguishable externally by an alveolar network which owes its origin to directional forces which the evaporating solution media exerts on the coagulating nitrocellulose aggregate. (The so-called Benard's vortex -reference 7). This characteristic structure is visible in fig.1. Whether and to what extent this nonhomogeneity influences the formation of equal-sized pores is as yet not clear. In the monograph by Jander and Zakowski (reference 4) An influence is first clearly denied (p.94, at another place, p.101) it is nevertheless completely under consideration (i.e. elsewhere it is considered very important p.101). One can no doubt observe the differences between the

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alveolar networks and the alveolar centers much better when wet. One occasionally obtains a filter in which the network is clearly raised and others in which it is sunk to form-series of slots. Therefore it appears desirable to take suitable measures for the making of completely compatible filters. As the first task in this direction, this work should clarify whether the alveolar net causes real difference in the porosity, as stated by Grabar and Loureiro (reference 5) in the dimensions of the nitrocellulose aggregate. This decision is very difficult to make with the light microscope since owing to the interrupted scattered reflection of the light, the surface contours are not clearly perceptible. In comparison the electronmicroscopic printing process (reference 11) permits illustration of the surface of such a filter. The electronmicroscope may be one of the few possibilities which furnishes us a deeper look into the mechanism of filtration and by use of the models which have been called upon for quantitative treatment of these very difficult problems - (reference 9), perhaps a choice can be made. Previously M.v.Ardenne (reference 10) had concerned himself with the ultramicroscopical study of the collodion filters; but his survey does not permit us to answer the problems raised here so that it seems appropriate to again carry out such an investigation with the improved methods.

We used a printing process recently developed by Helwig (reference 12) for reproducing the surface of the filter. This

represents an especially fortunate combination of an extraordinary high contrasting, structureless section reproduced in Tungstenoxide - and a cohering, similarly structureless Siliconoxide skin (reference 13).

The surface of the membrane filter to be studied is first obliquely "damped" - (i.e. a deposit is laid down through vacuum evaporation) tungstenoxide. By appropriate selection of the angles of "damping" one has the plastic effect of the impressions in hand. The thin and very sensitive section of tungstenoxide is then uniformly damped with siliconoxide in order to raise its mechanical durability.

The main demands which a deposition by vacuum evaporation brings with it are: 1) the membrane filter to be treated must be absolutely dry and 2) completely free of softeners (plasticizers ?) which become gaseous during the dampening process and thus cause distortions.

This was attained by repeatedly boiling the filters in distilled water and drying them in absolute ether. The surface of the filter was no way altered by this treatment. After dissolutions of the filter substance in methylocetate the deposited double membrane can be viewed in the ultramicroscope. Figures 2 and 3 reproduces that surface of a membrane filter (No. 60 of the Gröttingen Membrane filter Factory) which is oriented opposite to the filtration flow. The impression in figure 2 was prepared from the inside of the reticulum, that in figure 3 from the rim of the reticulum. One notes immediately a distinct difference between the average pore size in each field.

Whereas the pore diameter in the inside of the reticulum varies

between 0.15 and 1μ , it amounts to only about half as much (0.04 to 0.5μ) on the rim of the reticulum because of a much thicker packing. In order to obtain a filter of uniform pore size one must strive for a reticulum-free structure (reference 8). Such a filter has already been manufactured in Gottingen.

It should also be mentioned that an electronmicroscopic surface impression is naturally not sufficient to reach quantitative conclusions on the actual output of the filter. Indeed, after examination of a great number of sections one can say with certainty that particles with larger diameters than that of the largest aperture will be held back but one can not determine whether smaller particles will actually pass through, since the filter itself is more than 100 times as thick as the pores visible at the surface which are not continued as canals of equal cross section but instead points of smaller diameter can be shown. Therefore the data indicate that the filter such as that figured here, is completely impermeable to particles of a diameter greater than 0.4μ .

For his challenging interest in this work, we wish to thank Herr Dr. H. König.

German

C.R. Robins
Translator

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